US ARMY RDECOM

Procedures, Requirements and Challenges Associated with Analysis of Environmental Samples for Chemical Warfare Material (CWM)

TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.

DOD Environmental Monitoring Data Quality (EMDQ) Workshop

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Presentation Outline

1. What kind of laboratory is capable of performing CWM analysis?
2. What are some special requirements and challenges for screening samples?
3. What procedures are required to meet those requirements and challenges?
4. What are some project examples?

Army Organization

U.S. Army

U.S. Army Materiel Command (AMC)

U.S. Army Research, Development and Engineering Command (RDECOM)

U.S. Army Edgewood Chemical Biological Center (ECBC)

Chemical Biological Applications and Risk Reduction (CBARR)

Environmental Monitoring Laboratory (EML)

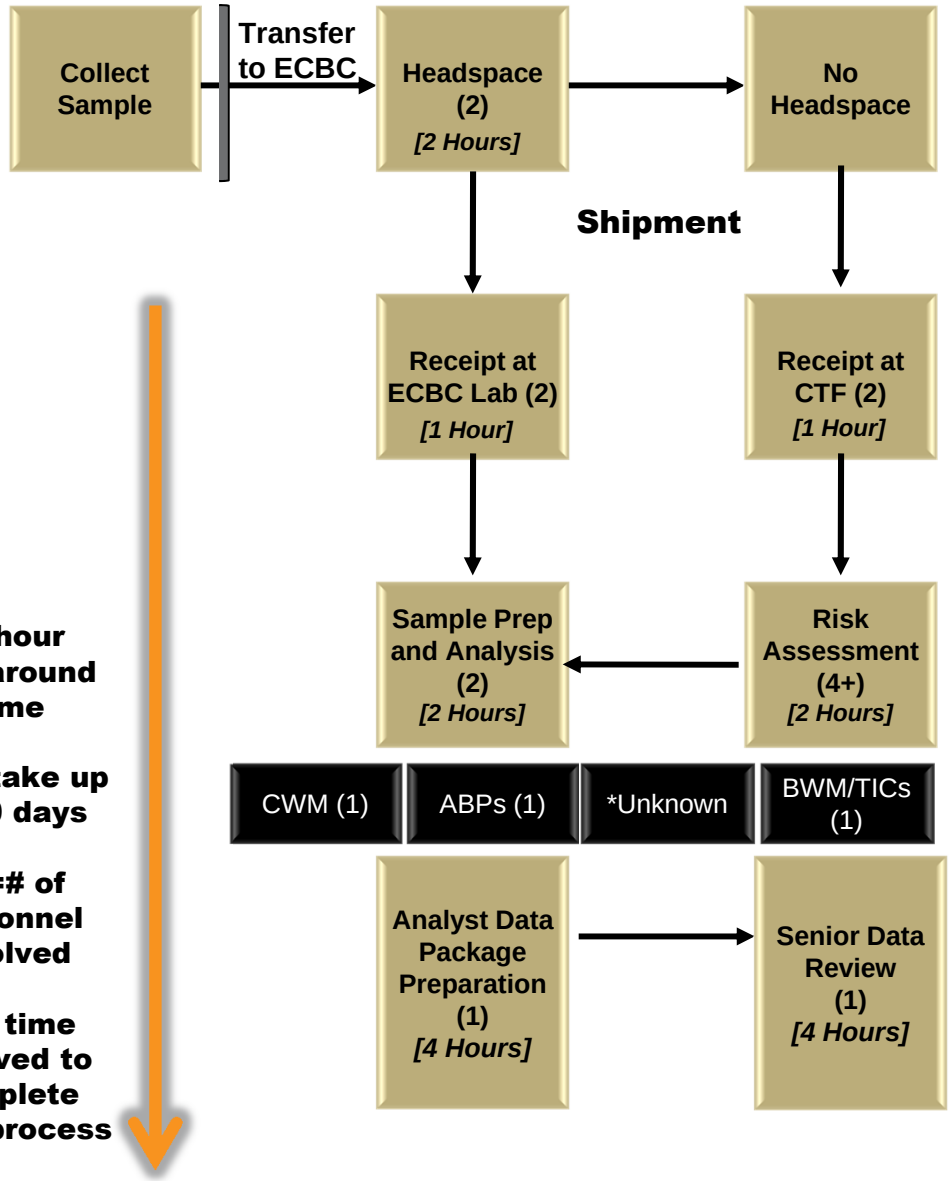
Environmental Monitoring Laboratory

- Services laboratory specializing in high-volume screening of samples from various environmental media for CWM, BWM and related compounds
- Air monitoring capabilities for CWM and industrial compounds
- Staff certified in C/B PRP
- Fixed and mobile laboratory services
- Large inventory of instrumentation and analytical equipment
- Registered Quality System w/ CASARM ISO 9001
- ISO17025 in BWM; working toward in CWM

Requirements and Challenges Associated with CWM Analysis

- **Rapid turnaround times (TAT) for results**
  - Impact to investigations
  - Must be completed prior to other testing methods
- **Should the analysis be performed in a mobile or fixed laboratory?**
  - Depends upon agents and degradation compounds of interest
  - Multiple screening procedures are required to test all the compounds of interest
  - Requirements for sample shipments
- **Meeting increased quality requirements**
  - Must be fully quantitative and legally defensible

Steps Involved in a Typical Environmental Analysis



Individual Methods ***[Up to 12 hours per method]***

24-hour Turnaround Time

***Can take up to 30 days**

( ) =# of personnel involved

[ ] = time involved to complete each process

What else is involved?

1. Individual Method Validation
2. Annual Method Verification
3. Analyst Certification
4. Agent Standard Testing
5. IOP/SOP Prep and Review
6. Instrument Maintenance
7. QC, Legally Defensible Data
8. Trained and Experienced Staff

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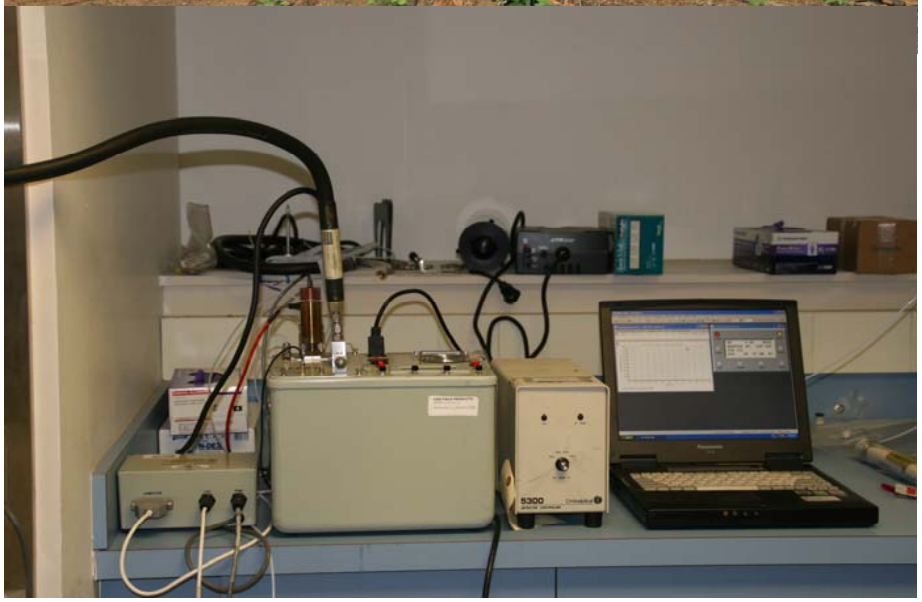
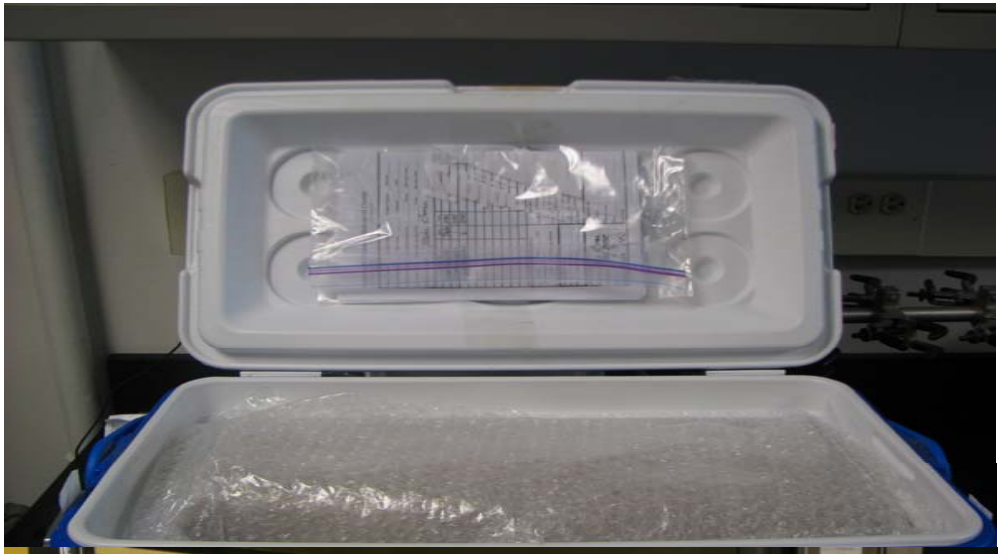
Vapor Screening of Environmental Samples

- Commonly referred to as “Headspace” or “Hotbox” procedure
- Heats samples inside a closed container to allow them to off-gas
- Container vapor is then sampled using MINICAMS or solid sorbent tubes
- Samples that are cleared below 1 VSL can be shipped commercially to the agent laboratory



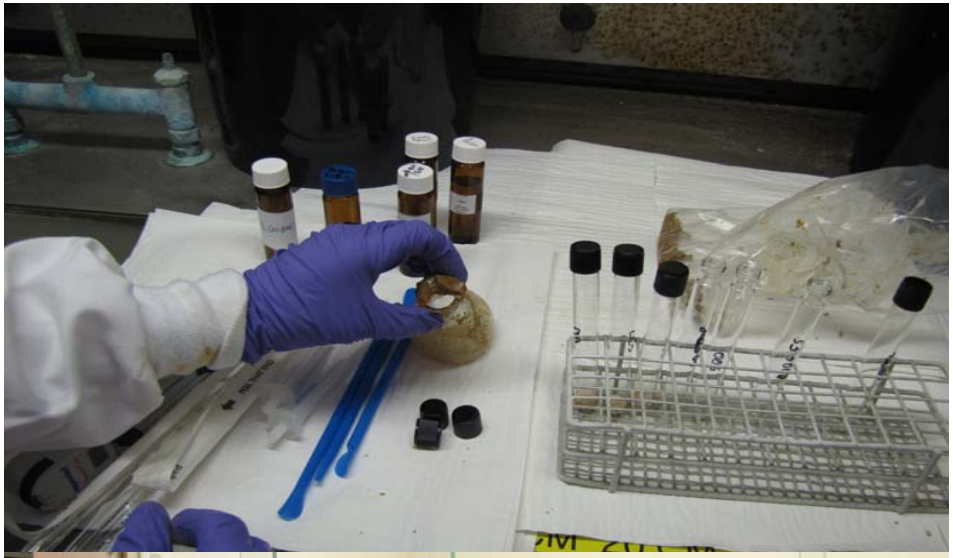
Receipt at Laboratory

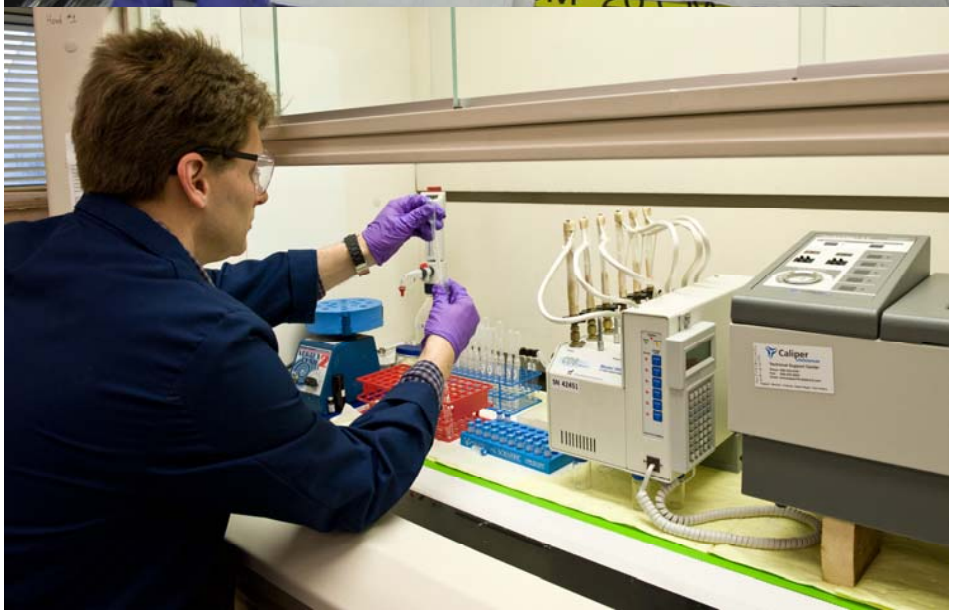
- Prior notification critical for receipt of samples
- Shipment must arrive on time via commercial carrier and COC correct
- Timing important so that instruments are calibrated and ready to start analysis
- Analysts are ready to log samples and begin sample preparation



Sample Preparation

- Samples are prepared in batches with typical QC
- All traditional CWM and some ABPs can be screened in one method
- Rest of the common ABPs can be screened in two to three or more methods
- Common solvent extraction procedures using surrogate and internal standards
- Utilizing isotopically labeled standards when available





EML Compounds of Interest

Procedure MT-08 Compound of Interest	Abbreviation
Bis(2-chloroethyl) sulfide~	Sulfur Mustard, H, HD
1,4-dithiane	N/A
1,4-oxathiane	1,4-thioxane
Bis (2-chloroethyl) ethylamine	HN-1
Tris (2-chloroethyl) amine	Nitrogen Mustard, HN-3
Dichloro(2-chlorovinyl) arsine	Lewisite, L
2-chlorovinyl arsenous acid *	CVAA
2-chlorovinyl arsenous oxide *	CVAO
Isopropyl methylphosphonofluoridate	Sarin, GB
Ethyl *N*,*N*-Dimethylphosphoramidocyanidate	Tabun, GA
O-Pinacolyl methylphosphonofluoridate	Soman, GD
Cyclosarin	GF
methyl-, S-[2-[bis(1-methylethyl) amino]ethyl] O-ethyl ester	VX

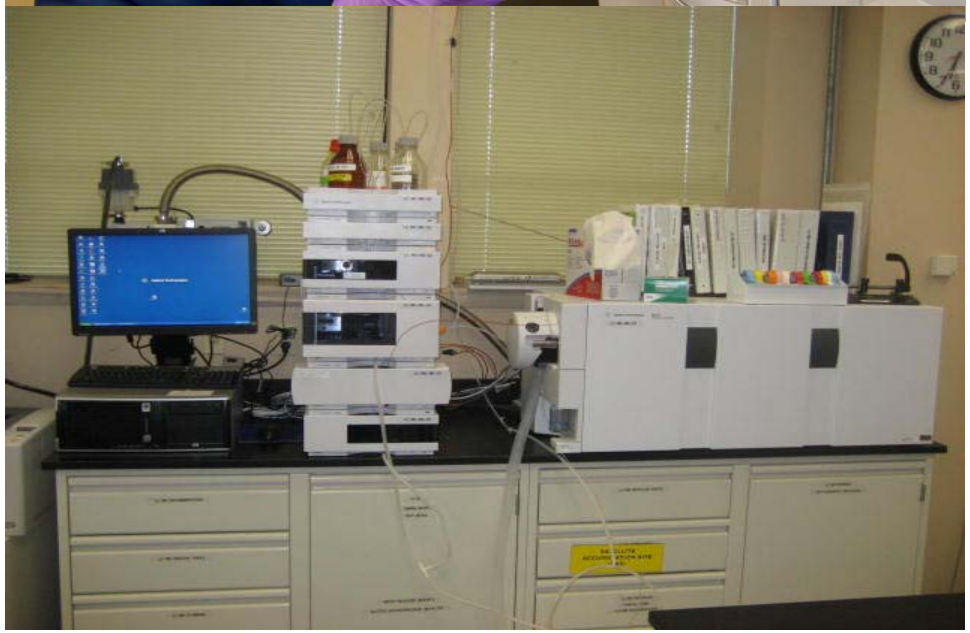
Procedure MT-57 Appendix IV Compound of Interest	Abbreviation
Diethanolamine	DEA
Triethanolamine	TEA
n-ethyl diethanolamine	EtDEA

Procedure MT-57 Appendix V Compound of Interest	Abbreviation
Thiodiglycol	TDG

Procedure MT-57 Appendix VI Compound of Interest	Abbreviation
Diisopropylmethyl phosphonate	DIMP
Dimethylmethyl phosphonate	DMMP
S-2-Diisopropylaminoethyl methylphosphonothioic acid	EA2192
Ethyl methylphosphonic acid	EMPA
Isopropyl methylphosphonic acid	IMPA
methylphosphonic acid	MPA
Pinacolyl methylphosphonic acid	PMPA

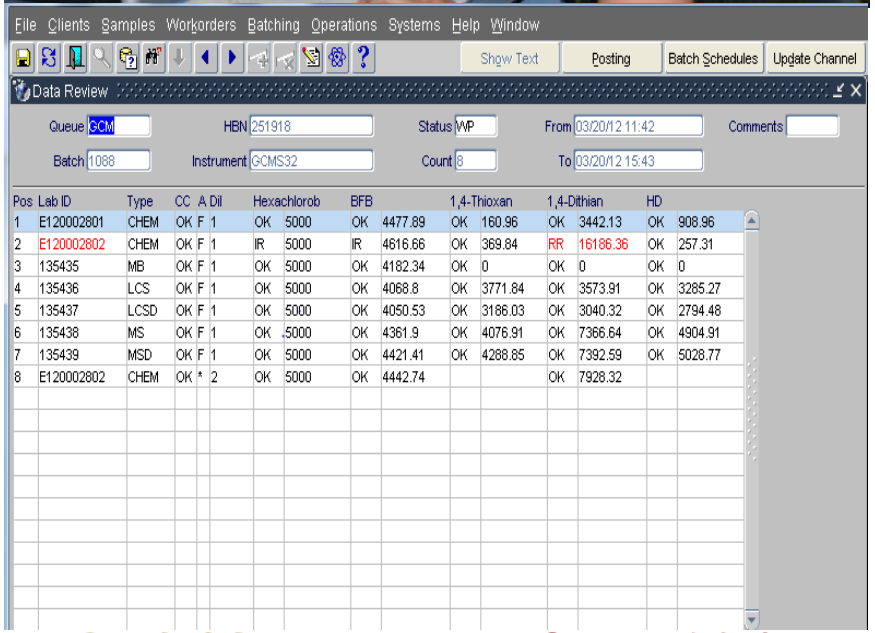
Sample Analysis

- Analysis methods are GCMS and LCMS-MS based
- GCMS methods uses five-point calibration
- LCMS-MS methods uses seven-point calibration
- Methods are validated annually
- Laboratory established QC limits for spiked samples updated annually
- Fifteen to 20-minute analysis time



Data Preparation, Review and Reporting

- Data package prepared by analyst the following morning dependent upon batch size
- Technical review performed by lab manager or senior staff
- QA/QC review
- Final report sent to the customer
- Split samples sent for further analysis
- Data package archiving for third party validation at Level IV
- Twenty-four to 48 hour TAT!

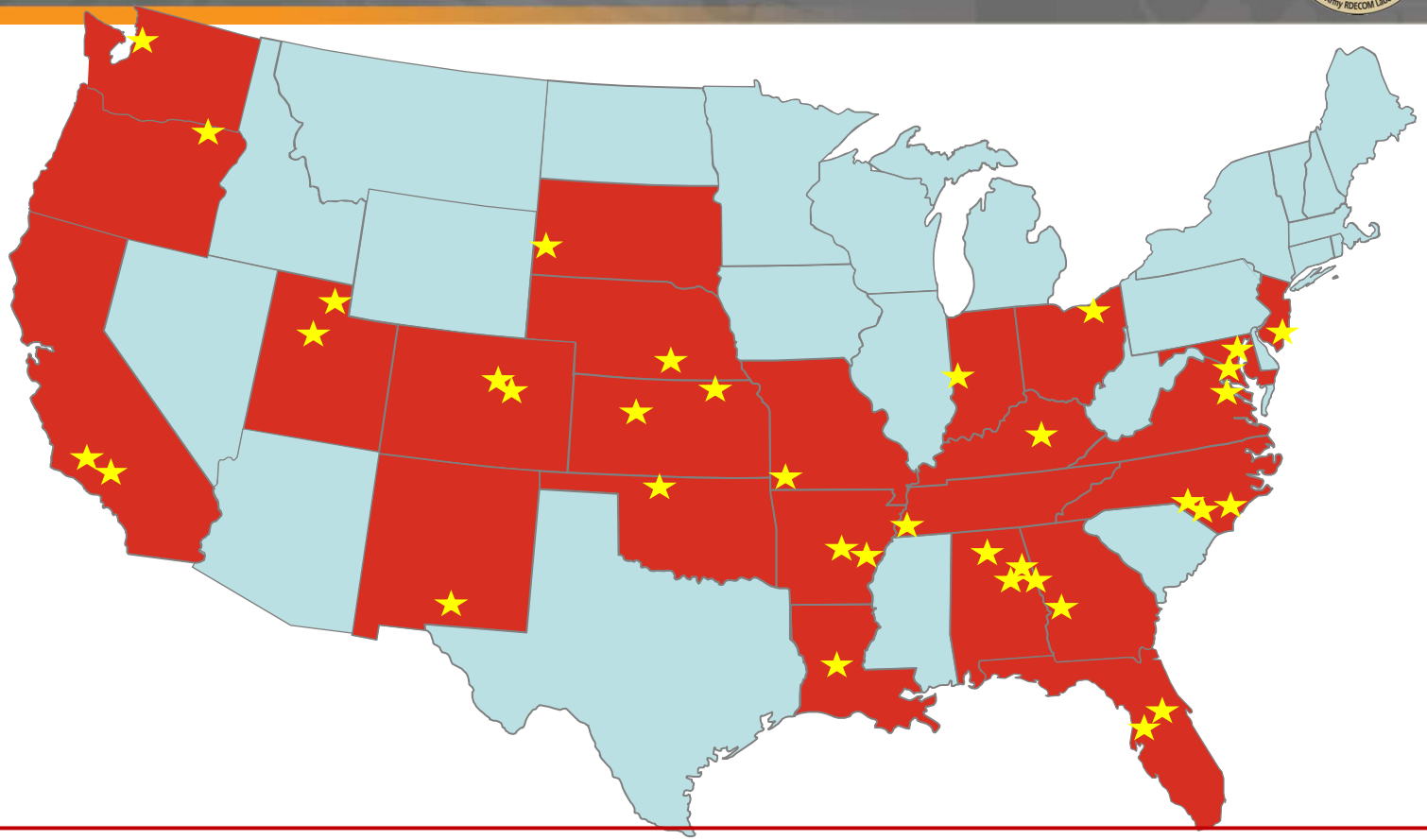


Pos	Lab ID	Type	CC	A	Dil	Hexachlorob		BFB		1,4-Thioxan		1,4-Dithian		HD	
1	E120002801	CHEM	OK	F	1	OK	5000	OK	4477.89	OK	160.96	OK	3442.13	OK	908.96
2	E120002802	CHEM	OK	F	1	IR	5000	IR	4616.66	OK	369.84	RR	16186.36	OK	257.31
3	135435	MB	OK	F	1	OK	5000	OK	4182.34	OK	0	OK	0	OK	0
4	135436	LCS	OK	F	1	OK	5000	OK	4068.8	OK	3771.84	OK	3573.91	OK	3285.27
5	135437	LCSD	OK	F	1	OK	5000	OK	4050.53	OK	3186.03	OK	3040.32	OK	2794.48
6	135438	MS	OK	F	1	OK	5000	OK	4361.9	OK	4076.91	OK	7366.64	OK	4904.91
7	135439	MSD	OK	F	1	OK	5000	OK	4421.41	OK	4288.85	OK	7392.59	OK	5028.77
8	E120002802	CHEM	OK	*	2	OK	5000	OK	4442.74			OK	7928.32		

Reporting Limits

Analyte	HBESL-Residential (Soil) (µg/kg)	HBESL-Industrial (Soil) (µg/kg)	MFWS (µg/L)	Laboratory Limits (Soil) (µg/kg)		Laboratory Limits (Aqueous) (µg/L)	
				MDL	LOQ	MDL	LOQ
H	10	300	47	2.0	10	2.0	10
1,4-Thioxane				17	100	19	100
1,4-Dithiane				18	100	20	100
HN-1				15	100	18	100
HN-3				15	100	20	100
L	300	3700	27	23.9	100	22.5	100
GB	1300	32000	4.0	15	100	19 (3.3)	100 (10)
GA	2800	68000	4.0	14	100	19 (3.2)	100 (10)
GD	220	5200	4.0	15	100	20 (3.3)	100 (10)
GF	220	5200	4.0	14	100	18	100
VX	42	1100	4.0	18 (0.021)	100 (0.064)	19 (0.018)	100 (0.053)

CONUS Projects



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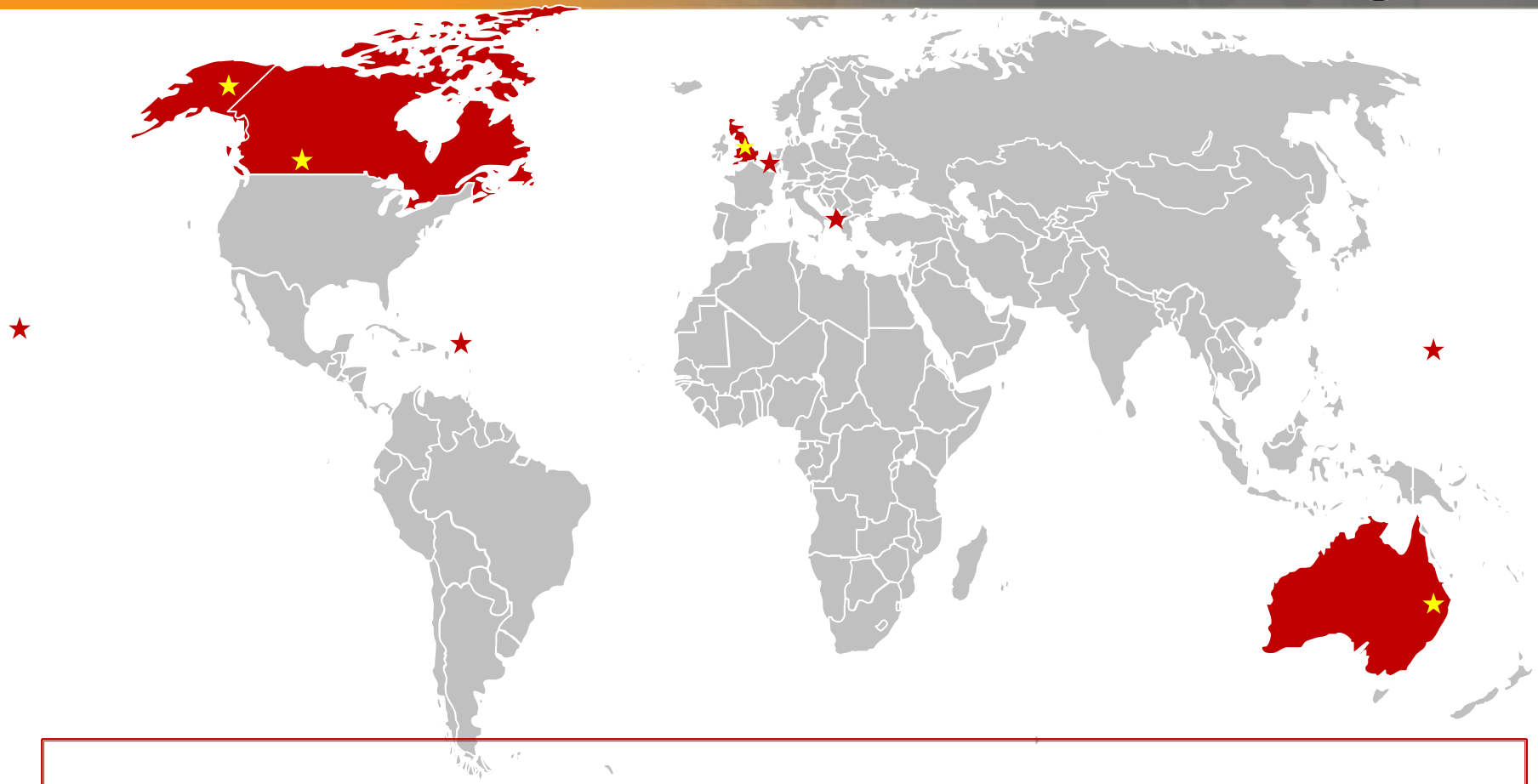
OCONUS Projects



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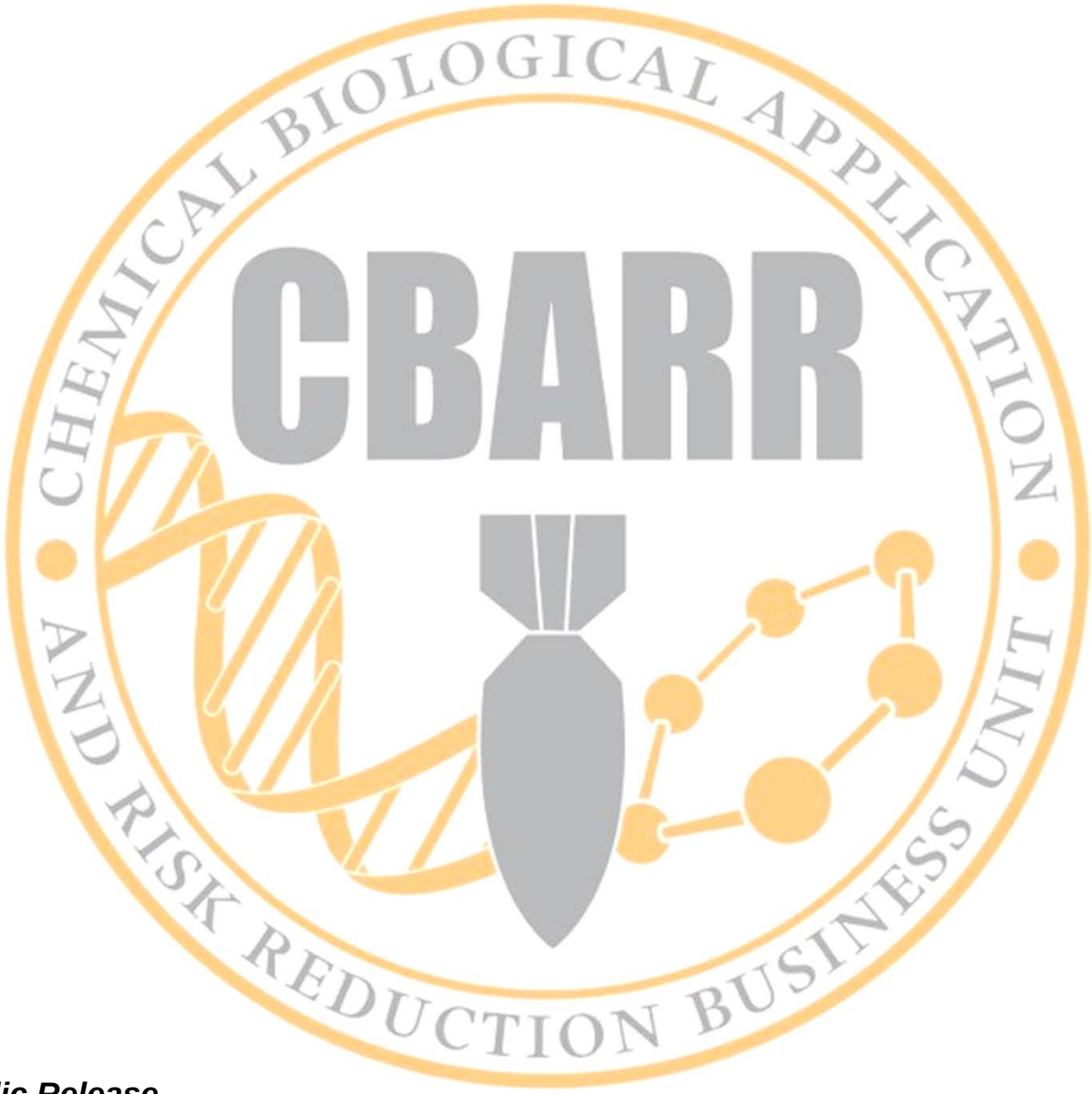
Questions



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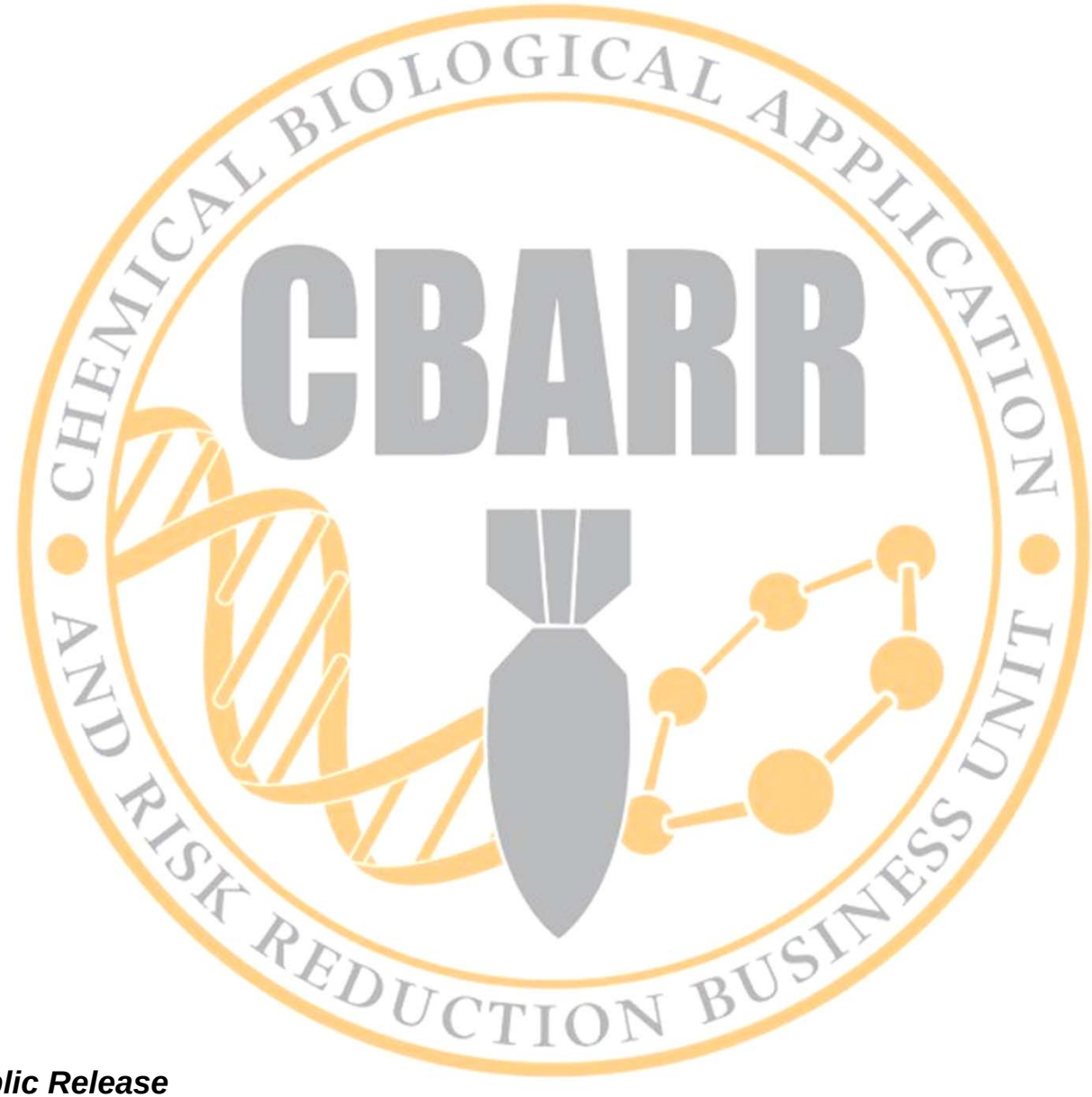
Backup Slides



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Reporting Limits

Analyte	Laboratory Limits (Soil) (µg/kg)		Laboratory Limits (Aqueous) (µg/L)	
	MDL	LOQ	MDL	LOQ
DEA	26	125	4.0	25
TEA	26	125	2.8	25
EtDEA	26	125	3.1	25

Reporting Limits

Analyte	Laboratory Limits (Soil) (µg/kg)		Laboratory Limits (Aqueous) (µg/L)	
	MDL	LOQ	MDL	LOQ
DIMP	20	60	2.6	12
DMMP	18	60	2.8	12
EA2192	51	150	6.9	21
EMPA	23	68	2.6	12
IMPA	40	120	7.7	23
MPA	38	180	7.7	37
PMPA	24	120	4.0	25

Reporting Limits

Analyte	Method of Analysis	Laboratory Limits (Soil) (µg/kg)		Laboratory Limits (Aqueous) (µg/L)	
		MDL	LOQ	MDL	LOQ
TDG	3	23	130	5.3	25